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EP 0315244 A2 **US 3714111 A**

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(54) Thermal insulant materials

(57) A thermally insulating plastics material, e.g. for lining the casing of a solid fuelled rocket motor, comprises an elastomer filled with a fibrous material which, on heating, degrades endothermically to carbon. Typically the filler comprises partially oxidised polyacrylonitrile, and the elastomer is EPDM, chlorosulphonate polyethylene, or a nitrile rubber/polyvinyl chloride mixture.

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THERMAL INSULANT MATERIALS

This invention relates to thermal insulant materials, and in particular to materials suitable for providing a thermally insulating barrier between the propellant charge and the casing of a solid fuel rocket motor.

A typical solid fuel rocket motor comprises a body or charge of propellant material disposed in a steel casing having an opening through which gas generated by the burning propellant is ejected. It is desirable to place a thermally insulating barrier between the propellant and the casing for two reasons. Firstly, the very high temperature and the gas ablative effects generated by the burning charge can burn through the casing and thus severely impair the operation of the motor. Secondly, the propellant gas is subjected to cooling via the thermally conductive casing this reducing the gas pressure available to operate the motor.

The provision of an insulant barrier effectively reduces the above problems. However, the selection of suitable materials has introduced further difficulties. For example, ceramic materials are very effective but are very difficult to manufacture. Plastics materials have therefore been proposed, but the thickness of conventional plastics for effective insulation severely reduces the volume available for the propellant charge.

The object of the invention is to minimise or to overcome the above disadvantages.

According to the invention there is provided a thermally insulant plastics material including an elastomeric matrix incorporating a fibrous filler material the filler material being in the form of short fibres and comprising a partially degraded polymer adapted to carbonise by an endothermic reaction or the application of heat to the plastics material.

Typically the fibrous filler comprises polyacrylonitrile. We have found that exposure of this material to intense heat causes transformation of the partially oxidised polyacrylonitrile (PAN) fibres to carbon fibres so as to provide a carbonaceous support for the thermally degraded plastics material. This maintains the thermal insulating properties of the material for a significant period of time following exposure to a heat source. We have also found that the transformation of the partially oxidised PAN fibres to carbon fibres is an endothermic process. The heat absorbed by this process provides a local cooling effect and further enhances the thermal insulating properties of the material.

Advantageously the composition further includes finely divided silica which provides a bulky refractory ash content and further improves the heat insulation effect.

A number of elastomer materials may be employed to form the thermoplastics matrix. For example, we have successfully employed ethylene propylene diene monomer, chlorosulphonate polyethylene and nitrile rubber/polyvinyl chloride. The cost of these materials is of particular advantage as, under high temperature conditions it undergoes an endothermic degradation process, similar to

that of the partially oxidised PAN fibre, thus further contributing to the effectiveness of the material as a thermal barrier.

The materials may be formed by compounding the polymeric material with the partially oxidised PAN fibres and a curing agent. Preferably the mix also incorporates finely divided silica. The compounded material is formed into a sheet and is then heated to effect curing. Typically the sheet is between 1 and 2 mm in thickness. Advantageously the PAN fibres are about 2 mm in length and about 10 microns in diameter.

The materials are of particular application as thermal insulants for solid fuelled rocket motors. For this purpose the material may be worked on a two-roll mill before being calendered to form a sheet approximately 2um thick. The calendered sheet may be used to line a tubular motor casing, a number of layers being used to build up the required thickness. A plastics bladder is inserted and inflated to apply pressure to the liner which is then cured, e.g. by heating in an autoclave for about 1 hour at a temperature of 140 to 160°C.

To simulate exposure to a burning rocket propellant, we have tested these materials by exposure to an oxygen/acetylene flame by the method described in ASTM E285. As a result of these tests we have found that the thermal insulant materials described herein retain their heat insulating properties for periods of time significantly longer than those exhibited by conventional materials.

To demonstrate the invention a number of plastics compositions have been prepared and tested by the method of ASTM IE 285. The results of these tests are summarised in the following example.

EXAMPLE

A number of polymeric compounds were prepared using EPDM, a chlorosulphonate polyethylene and a PVC based nitrile rubber, the batch compositions being summarised below. The compounds were mixed in an Internal mixer using a fill factor of 0.9 - 0.93. A conventional mix cycle was used with the polymer and process aids being added first. The silica and fibres were then incorporated into the polymer matrix, finally the cure system was added. The temperature of the mixing commenced at between 75 and 80°C and the batch was dumped before the temperature had reached 120°C. The dumped material was then consolidated on a two-roll mill to form a homogeneous hide.

For test purposes each hide was cut into small pieces and compression moulded for 60 minutes at 150°C using a simple plate mould to form sheets nominally 1.5 and 6mm thick.

Samples of the sheets were subjected to flame testing by the method described in ASTM E285, the result being summarised below. For comparative purposes similar testing was carried out on two conventional ceramic filler materials.

Batch No. ML043

EPDM based (Ethylene Propylene Diene Monomer)

	Parts by weight	
Nordes N2760 (EDPM)	97	
Hypalon 40	3	
Zinc Oxide	5	)
Polyethylene Oxide	3	)
Tetrone A	2.5	(Cure System
Stearic acid	1	)
Vulcanford ZDEC	0.5	)
Coumarone Resin	5	Process Aid
Microsil ED (silica)	40	
Oxidised PAN fibres (2mm)	12	

Batch No. ML062

Hypalon Based (Chlorosulphonate Polyethylene)

	Parts by weight	
Hypalon 40	100	
Nordel N2760	3	
Maglite D (MgO)	4	)
Pentaerythritol	3	(Cure System
Tetrone A	2	)
Coumarone Resin	2.5	
Microsil ED (Silica)	35	
Oxidised PAN fibres 2mm	12	
Ucarsil DSC RC1/50	10	

Batch No. 057

Nitrile Rubber.PVC based

	Parts by weight	
Perbunan NVC/70	100	
Renocure	2.5	)
Vulcacit C2/EG	1.5	(Cure System
Vulcacit Thiuram C	0.2	)
Microsil ED	35	
Ucarsil DSC RC1/50	10	
Oxidised PAN fibres 2mm	12	

COMPARATIVE ABLATION RESULTS OF THERMAL INSULANT MATERIALS

	FORMULATIONS UNITS	CL2759	CL7225	ML043 EPDM	ML057 NBR/PVC	ML062 Hypalon
		(currently used materials)		(BNRE developed materials)		
Density	kg/m^3	1760	1094	1098	1243	1317
Insulation to density performance/80	$S.m^2/kg$	0.99	0.07	6.45	6.48	6.60
" /180	$S.m^2/kg$	1.00	0.27	7.67	7.49	7.26
" /380	$S.m^2/kg$	1.03	2.56	7.87	7.88	7.62
Average time to burn-through	S	13.8	20.5	60.7	69.6	72.3
Average erosion rate	$m/s \times 10^{-4}$	5.044	3.316	1.140	1.004	0.966
Average insulation index /80	S/m	1734	75	7080	8056	8699
" /180	S/m	1763	292	8418	9308	9556
" /380	S/m	1806	2796	8639	9790	10036
Average time to 80	S	12.0	0.5	48.9	56.0	60.7
" 180	S	12.2	2.0	58.1	64.7	66.7
" 380	S	12.5	18.7	59.6	68.0	70.0

These results illustrate the effectiveness of the materials described herein as thermal insulants.

CLAIMS

1. A thermally insulant plastics material including an elastomeric matrix incorporating a fibrous filler material the filler material being in the form of short fibres and comprising a partially degraded polymer adapted to carbonise by an endothermic reaction or the application of heat to the plastics material.

2. A thermally insulant plastics material as claimed in claim 1, wherein the fibrous filler comprises partially oxidised polyacrylonitrile.

3. A thermally insulant material as claimed in claim 1 or 2, wherein the elastomer comprises ethylene propylene diene monomer, chlorosulphonate polyethylene or a nitrile rubber/polyvinyl chloride mixture.

4. A thermally insulant material as claimed in claim 1, 2 or 3, and incorporating finely divided silica.

5. A thermally insulant material as claimed in claim 2, 3 or 4, wherein the fibrous filler comprises about 12 wt % of the material.

6. A thermally insulant material as claimed in claim 1 and substantially as described herein.

7. A solid fuel rocket motor lined with an insulant material as claimed in any one of claims 1 to 6.

Amendments to the claims have been filed as follows

1. A plastics composition adapted to provide thermal insulation at elevated temperatures, the composition comprising an elastomeric matrix incorporating a fibrous filler material in the form of short fibres and consisting of a partially degraded polymeric material, wherein the filler is such that, when the plastics composition is in use exposed to a heat source causing thermal degradation of the elastomeric matrix, the filler material carbonises via an endothermic process whereby to absorb heat and to provide a carbonaceous support for the thermally degraded elastomeric matrix.

2. A plastics composition as claimed in claim 1, wherein the fibrous filler comprises partially oxidised polyacrylonitrile.

3. A plastics composition as claimed in claim 2 wherein said films are about 2mm in length and about 10 microns in diameter.

4. A plastics composition as claimed in claim 1, 2, or 3, wherein the elastomer comprises ethylene propylene diene monomer, chlorosulphonate polyethylene or a nitrile rubber/polyvinyl chloride mixture.

5. A plastics composition as claimed in claim 1, 2, 3 or 4, and incorporating finely divided silica.

6. A plastics composition as claimed in claim 2, 3 4 or 5, wherein the fibrous filler comprises about 12 wt % of the material.

7. A plastics composition as claimed in claim 1 and substantially as described herein.

8. A solid fuel rocket motor lined with plastics composition as claimed in any one of claims 1 to 7.

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Relevant Technical fields

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Databases (see over)

(i) UK Patent Office

(ii) Online: WPI, WPIL

Documents considered relevant following a search in respect of claims 1-7

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	EP-A2-0315244 (DOW) page 3 line 44 to 51, Example 1	1-5
A	US-A-3714111 (ECONOMY) column 3 line 32 to to column 4 line 21	1